



Facile controllable synthesis of magnetite nanoparticles via a co-precipitation approach

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Abstract

Magnetite (Fe_3O_4) nanoparticles were controllably prepared by a co-precipitation approach using two precipitating agents such as sodium hydroxide and ammonium hydroxide at relatively low temperature ($40\text{ }^\circ\text{C}$). The as-prepared samples were characterized by using X-ray diffraction (XRD), Fourier transform infrared (FT-IR), scanning electron microscope (SEM), transmission electron microscope (TEM) and vibrating-sample magnetometer (VSM) techniques. The results revealed that ammonium hydroxide produced magnetite nanoparticles with smaller particle size (14 nm), compared to those prepared (22 nm) using sodium hydroxide precipitating agent. In addition, we found that the saturation magnetization of the produced magnetite nanoparticles was influenced by the obtained particle size. The determined saturation magnetization of the generated magnetite nanoparticles was found to be ca. 61.3, 65.1 emu/g for those prepared using NaOH and NH_4OH precipitating agents, respectively.

Keywords: Magnetite nanoparticles; co-precipitation approach; sodium hydroxide; ammonium hydroxide; vibrating-sample magnetometer.

1. Introduction

Magnetite (Fe_3O_4) nanoparticles have lately established a lot of attention because of their diverse physical and chemical features at the nanoscale [1]. Magnetic and electric characteristics of magnetite (Fe_3O_4) nanoparticles are very fascinating [2]. The cubic inverse spinel structure of Fe_3O_4 is unique. It might be an n-type or p-type semiconductor. It has the minimum resistivity between iron oxides because its tiny band gap (0.1eV) [3]. Because of the alternating Fe^{2+} and Fe^{3+} lattice interrupted by oxygen atoms that allow for electrical interaction, magnetite is ferromagnetic [4]. Furthermore, surface alteration of magnetite nanoparticles can raise not only their solvent stability, but also their interfacial features and avoid particle aggregation [5]. Magnetite nanoparticles have exclusive properties, including decent magnetic, electric, great surface area, broad surface-to-volume ratio, and simple separation under an external magnetic field, catalytic, respectable chemical activity and susceptibility to oxidation, biocompatibility and low toxicity properties [6]. They are widely used in bio separation, catalysis, industrial processes, color imaging, magnetic recording media, soft magnetic materials, ferrofluid, Protein immobilization spintronic, and, most importantly, biological sciences because to their features [7]. The use of magnetite

nanoparticles as carriers for protein immobilization is one of the most common and commonly utilized applications. [8]. Due to features such as the absence of flocculants created and the high effluent amount managed in a less period of time, adsorbents magnetic separation has established its applicability as a viable approach for wastewater treatment. The magnetic materials are recommended as appropriate candidates for dye sequestration with quick separation and increased water decontamination efficiency. Coated magnetite nanoparticles intended to remove various water pollutants have been the subject of several scientific studies. In other investigations, iron-based nanoparticles achieved using green synthesis methods utilizing extracts derived from agro-wastes as low-cost bio reducing agents were used to eliminate diverse colored pollutants from waters [9].

In bioanalytical operations, magneto-optical switches, magneto optical sensors, modulators, optical circulators, and optical isolators, transparent magnetic materials show a substantial role [10].

Lastly, there has been a lot of interest in creating transparent magnetic materials with outstanding magnetic, optical, mechanical, and thermal characteristics [11]. Spin coating a solution of magnetite fine particles on glass substrates, accompanied by drying at $80\text{ }^\circ\text{C}$ and solidifying

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at 200 °C in a magnetic field, results in a translucent magnetic thin polyvinyl alcohol film [11]. By covalently attaching liquid crystals to siloxane backbones and then coupling them to dopamine-functionalized Fe₃O₄ nanocrystals, Hyon Min Song created transparent magnetic composites [12].

Due to its remarkable magnetic characteristics, magnetite Fe₃O₄ is one of the most valuable industrial materials. Due to the lack of any hazardous elements for biological tissues, Fe₃O₄ appears to be the best choice for therapeutic applications. Magnetite nanoparticles have the capability for attaching the cell wall of bacteria. Lipopolysaccharides, phospholipids, proteins, and lipoteichoic acid are all crucial component of the bacterial membrane and may be found on the bacteria's surface. These biomolecules' functional group aids bacteria adhesion to diverse surfaces and multiplication [13]. Because of its superparamagnetic characteristics and lack of toxicity to humans, magnetite is becoming increasingly attractive. When these nanoparticles come into touch with air, they lose their magnetic and monodispersibility [14]. The magnetite Fe₃O₄-C18 composite nanoparticles were accomplished and used to clean and enrich 14 various organophosphorus species found in fruits and vegetables [15]. Magnetite (Fe₃O₄) has been extensively reported to aid in the creation of hydrogen during dark fermentation [16]. Nevertheless, with the optimum dosage concentration, the efficiency of fermentative bio-hydrogen generation will be increased. The effect of (Fe₃O₄) on bio hydrogen product was investigated [17]. Magnetite can also be used to improve the fabric's surface morphology, chemical and phase components, crystalline structure, magnetic behavior, thermal stability, and mechanical characteristics. [18]. There are several diverse approaches for fabrication of magnetite nanoparticles like co-precipitation method [19], sol-gel method [20], electron beam lithography [21], gas segment deposition [22], oxidation method [23], reduction method [24], hydrothermal method [25], solvothermal method [26], thermal decomposition method [27], flow injection method [28], electrochemical method [29], vapor phase method [30], supercritical method [31], sonochemical decomposition method [32], nonreactors method [33] microbial method [34], electrochemical synthesis [35], micro emulsion techniques [36], hydrolysis [37], and microwave assisted synthesis [38]. The co-precipitation approach in aqueous media is a favorable method for magnetite synthesis since the synthesis process is easy and the environmental impact is minimal. The co-precipitation method is a simple way to make magnetite nanoparticles from an aqueous iron salt (Fe²⁺, Fe³⁺) solution by simply adding a base at room temperature in an

2.3. Characterization

The x-ray diffractometer (Bruker; model D8 advance) with monochromated Cu-K α radiation ($\lambda = 1.54178 \text{ \AA}$) was applied to identify the crystallinity and phase purity of as-prepared products. The morphology and particle size of the prepared samples were tested by a higher resolution transmission electron microscope (HR-TEM) (JEM - G20 F) with 200 KV accelerating voltage and FE-SEM pictures were gathered on a field emission scanning electron microscope (FE-SEM) associated with a microscope

inert environment. The co-precipitation technique produces no hazardous intermediates or solvents, requires no precursor complexes, and operates at temperatures below 100 °C. Because of its ability to be scaled up, reproducibility, and environmentally acceptable reaction conditions, this technique has been acknowledged for its industrial usefulness [39]. It does, however, produce particles with a wide size distribution and large particle size [40]. Therefore, in the present work, magnetite nanoparticles were successfully prepared through a co-precipitation approach. In this light, iron(III) sulfate (Fe₂(SO₄)₃·5H₂O), and iron(II) sulfate (FeSO₄·7H₂O) were employed as an iron precursor. We studied the controllable preparation of magnetite using two different precipitating agents such as sodium hydroxide and ammonium hydroxide. The effect of precipitating agent on morphology, particle size, and saturation magnetization of the as-prepared magnetite nanoparticles was investigated. Diverse tools were used to investigate the prepared magnetite nanoparticles such as XRD, IR, SEM, TEM and VSM.

2. Materials and Methods

2.1. Chemicals and materials

Ferric sulfate pentahydrate (Fe₂(SO₄)₃·5H₂O) and ferrous sulfate heptahydrate (FeSO₄·7H₂O) were purchased from Alpha Chemika, India. Ammonium hydroxide (25% NH₄OH) and sodium hydroxide were acquired from El-Nasr pharmaceutical chemicals company, Egypt. All chemicals were applied as supplied without further purification.

2.2. Preparation of magnetite (Fe₃O₄) and other nanoparticles

In a typical co-precipitation method: 2.02 g of ferric sulfate pentahydrate and 1.41 g of ferrous sulfate heptahydrate were dissolved in 75 mL distilled water under magnetic stirring at temperature 40 °C until complete dissolution. To this, sodium hydroxide solution (2M, 25 mL) was added dropwise under magnetic stirring until reaching a value of pH from 9 to 12. The mixture was then cooled to room temperature. The black precipitate (magnetite) was collected through centrifuging at rotation 3000 rpm. The product (magnetite) was washed times with deionized water till reaching a value of pH 7.0 and then dried for 8 hours in an electric oven at temperature 80 °C. At the same circumstance, this procedure was repeated using ammonium hydroxide solution (25 %) as a precipitation agent instead of sodium hydroxide.

(JEOL JSM-6390F). The function group and magnetite formation were recognized by applying an FT-IR spectrometer (model Tensar 27, Bruker) from 4000 to 400 cm⁻¹ rang. Saturation magnetization of two magnetite sample were assessed using vibrating sample magnetometer (VSM, Lake Shore model 7410) from 25000 to -25000 Oe and 0 to 90 emu/g.

3. Results and Discussion

3.1. X-ray diffraction

Figure 1 displays the XRD patterns of the as-prepared magnetite nanoparticles synthesized by using aqueous solutions of sodium hydroxide (a) and ammonium hydroxide (b). The characteristic reflections of the synthesized magnetite nanoparticles appeared at 2θ values of 30° , 35° , 43° , 53° , 57° , and 62° , corresponding to crystalline lattice planes of (220), (311), (400), (422), (511), and (440), respectively, which are in accordance to the JCPDS card No. 88-0315 [41]. The crystal phase structure of the magnetite is cubic and no characteristic peak of impurities are detected. The average crystallite size of the prepared magnetite nanoparticles; sample (a) and sample (b), was estimated by using Scherer's equation [42] and it was found to be 22 and 14 nm, respectively.

$$D = \frac{K * \lambda}{\beta * \cos \theta}$$

Where: D is the mean size of the crystallite (nm), K is the shape factor of the crystallite (dimensionless), λ is the x-ray wavelength (nm), β is the line broadening at half the maximum intensity (FWHM) (radians), and θ is the x-ray diffraction angle (degrees). Interestingly, the results revealed that both precipitating agents generated pure magnetite nanoparticles; however, ammonium hydroxide precipitating agent gave magnetite nanoparticles with smaller crystallite size.

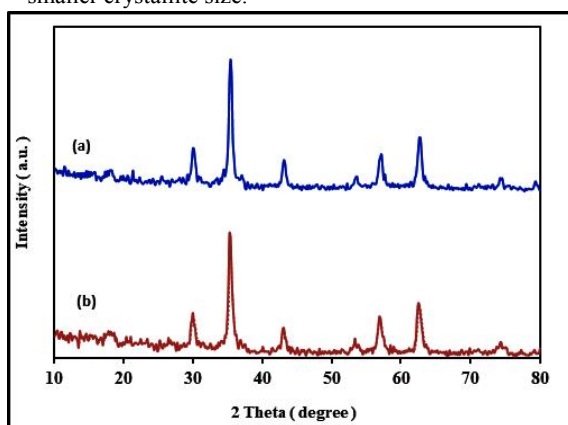


Figure 1: XRD patterns of magnetite nanoparticle samples prepared by using sodium hydroxide (a) and by ammonium hydroxide (b) precipitating agents.

The surface morphological structure of the as-prepared magnetite nanoparticles were examined using the scanning electron microscopy technique (Figure. 3). The SEM images of the magnetite products exhibited that both samples are composed of spherical particles. However, the magnetite nanostructure product prepared using ammonium hydroxide precipitating agent was more homogenous and regular sphere particles (Figure 3(a)). While the magnetite

3.2 The FT- IR spectra

The chemical structures of the as-prepared magnetite samples were identified by using FT-IR spectroscopy in range 4000 to 400 cm^{-1} . The FT- IR spectra of the magnetite products are presented in Figure 2. For samples, it can be seen that the broad characteristic band at 3400 cm^{-1} could be assigned to O-H stretching vibration of physically adsorbed hydrogen bonded water molecule [43-45]. Moreover, the spectra also revealed an absorption band at 1631 cm^{-1} , which can also be correlated to the bending vibration of bonded water molecules exist the iron oxide lattice [46]. The vibrational absorption bands at 558 cm^{-1} and 667 cm^{-1} , are attributed to the stretching vibration modes of the Fe-O bonds in tetrahedral and octahedral sites respectively [41, 47, 48].

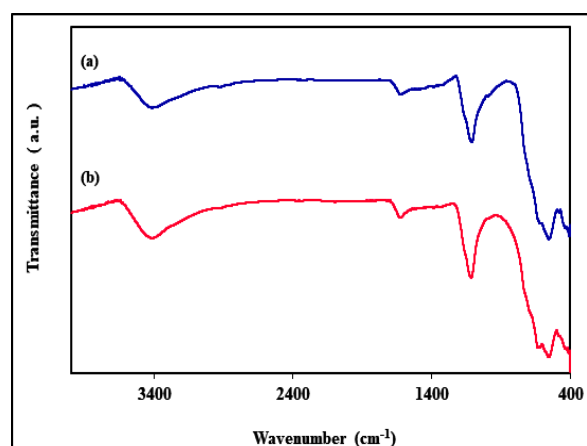


Figure 2: FT-IR spectra of magnetite nanoparticle samples prepared by using sodium hydroxide (a) and by ammonium hydroxide (b) precipitating agents.

product produced by sodium hydroxide precipitating agent consisted of spherical, cubic, and irregular particles (Figure 3(b)).

3.3 Morphological studies of prepared sample

In addition, the microstructure of the magnetite products was inspected by using transmission electronic microscopy technique as displayed in Figure 4. Inspection of the TEM images of the products revealed the magnetite nanoparticles prepared by employing sodium hydroxide was composed of spherical, cubic, and irregular particles with an average particle size of 20 nm. While the magnetite sample prepared by using ammonium hydroxide was composed of spherical and irregular particles of particle size of 12 nm. The estimated particle sizes of both products from TEM were compatible with those determined from the XRD results.

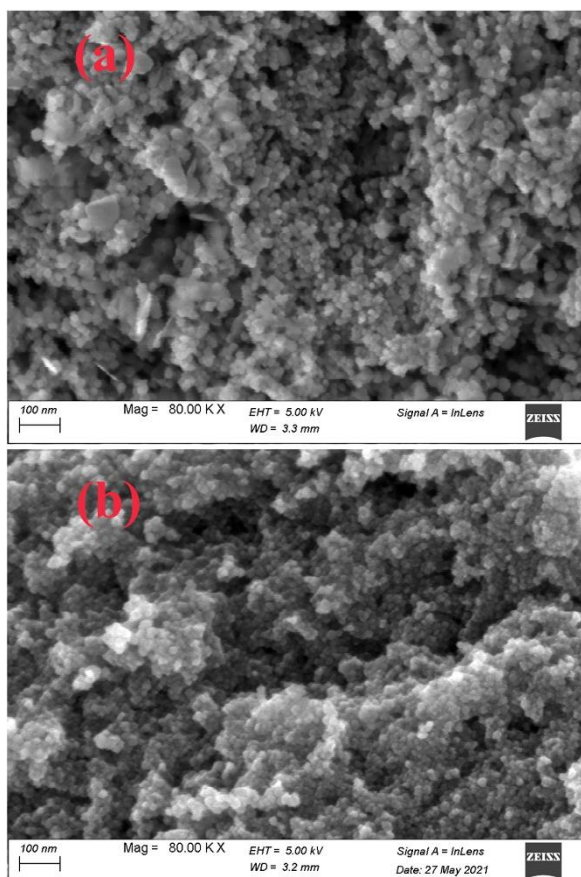


Figure 3: SEM images of magnetite nanoparticle samples prepared by using sodium hydroxide (a) and by ammonium hydroxide (b) precipitating agents.

It is notable that the magnetite nanoparticles synthesized using ammonium hydroxide was more homogenous and dispersed compared to those prepared using sodium hydroxide precipitating agent. Consequently, NH_4OH was opted as the optimum precipitating agent for our current preparation method for magnetite nanoparticles.

3.4 Magnetic properties of prepared samples

The magnetic characteristics of the as-prepared samples were investigated using vibrating sample magnetometer (VSM) and displayed in (Figure. 5). The saturation magnetization of the magnetite produced is approximately 61.3 emu/g for sample synthesized using NaOH and 65.1 emu/g for sample attained from NH_4OH at the similar conditions. Magnetic characterization can also be influenced by the size of magnetite nanoparticles and this is in accordance with the published data [49].

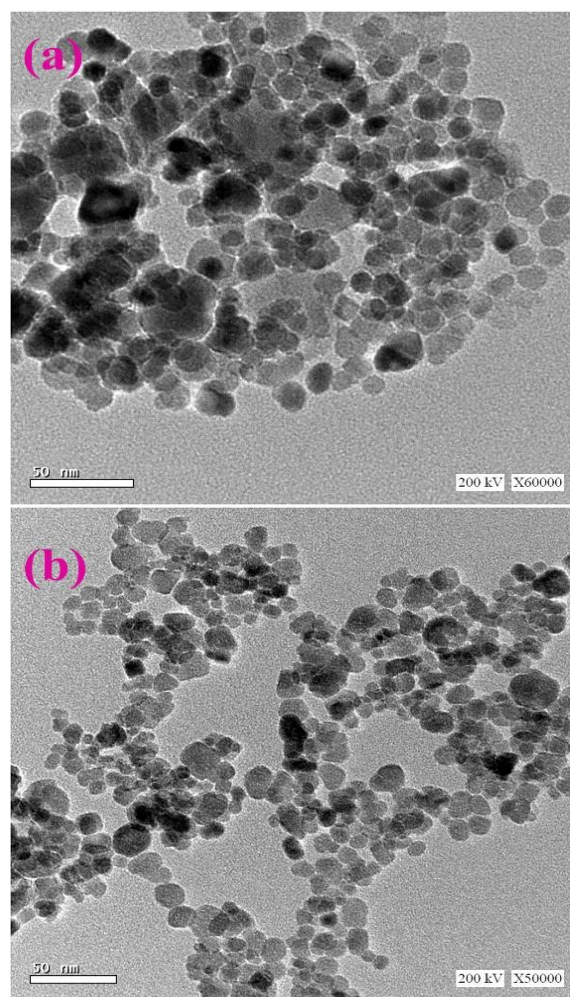


Figure 4: TEM images of magnetite nanoparticle samples prepared by using sodium hydroxide (a) and by ammonium hydroxide (b) precipitating agents.

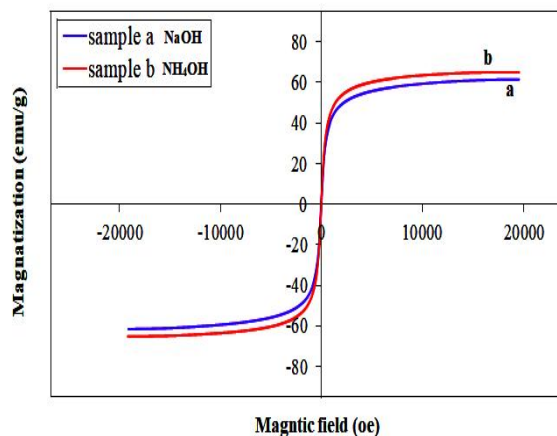


Figure 5: VSM of magnetite nanoparticles prepared by sodium hydroxide (a) and ammonium hydroxide (b) precipitating agents.

3. Conclusions

In the current work, magnetite (Fe_3O_4) nanoparticles were controllably prepared in great yield by co-precipitation at relatively lower temperature using two precipitating agents; NaOH and NH_4OH . The results revealed that ammonium hydroxide precipitating agent was the optimum precipitating agent for obtaining magnetite nanoparticles with smaller particle size and greater saturation magnetization value. This preparation approach could shed light on synthesis of magnetite with different particle sizes for different applications.

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